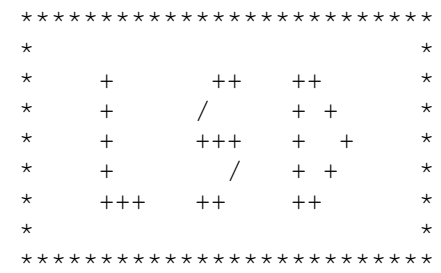
More Stuff.

How to make LSD

> So just _how_ does one make LSD?

HOW TO MAKE



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LSD : d-lysergic acid diethylamide

OK This file is about LSD.. I will detail some methods of making it from chemicals and from seed..
It should be noted that this file is for informational purposes only.. :) :)

HISTORY

LSD was first taken in its pure form by Dr. Albert Hoffman, a 37 year old Swiss chemist who worked at the Sandoz Pharmaceutical labs in Basel. In April 1943 he absorbed a drop of the 25th alkaloid solution (LSD-25) onto his finger by accident and noted that life had a "pleasant, fairy-tale quality". On April 19th he deliberatly swallowed some more, beginning with the tiny dose of 250 micrograms, or 25 millionths of a gram, a dose so small that no other drug known produces effects at these levels (200 micrograms is now considered a standard dose of LSD), and unbelivably to him he started tripping out.. Sandoz did further tests and these confirmed the enormous psychoactive potential for the drug..

The US army did huge experiments with LSD, testing it and its eight known deriatives as an incapitating agent for use in warfare, as well as testing it for possible uses in reversing brainwashing of soldiers.. There were secret tests done as well in which soldiers were unknowingly dosed and observed, also many were done in which they were knowingly dosed, and the films of these disorientated soldiers in a wartime situation were shown to demonstrate the great potential for LSD. Anyone outside the military can really only guess at the extent o f testing hidden from the public. The Russian scientists experimented with its warfare potential as well as parapsychological uses of LSD. The Weather Underground alledgedly held acid sessions to see if they had been infiltrated by an informer. The medica l profession also

latched onto LSD, testing it for possible uses in rehablilitating psychotics and schitsofrenics with some positive results. Similar good results were recorded for people with heavy sex hang-ups, people addicted to drugs, and psychopaths.

FORMS OF ACID

ACiD is normally sold in trips, little blotter paper tabs about 1cm big although the size varies.. It can also be liquid, crystalline in powder form or in tablets of any description eg- microdots.. The standard dose is around 200 mics but the strength of the ACiD will vary enormously of course, as will the quality of the high. The diffent types of LSD eg (LSD-25, LSD-21, LSD mirror 21) all give vastly different trips and each one is of course variable. I think that the crystalline and the liquid forms are the purest.

MAKING LSD

PT 1 - EXTRACTING LYSERGIC ACID FROM SEED

First get your seeds of preferably W1oodrose (argyria [hawaiian baby woodrose]) variety but Convolvulaceae, Rivea, or Ipomoea will do.. The overall yield with this method is low, however it can be done with easily obtained chemicals.. NOTE - the following procedure requires some knowledge of lab techniques and unless you know what you are doing you could easily blow yourself up, or poison you and your friends if the final product is imperfect.. Proceed with caution..

Finely grind seeds and add $NaHCO_3$. Extract with ethyl acetate by soaking about one day. Filter and extract the ethyl acetate with tartaric acid solution. Basify the extract with $NaHCO_3$ and extract with ethyl acetate. Dry and evaporate the ethyl acetate to get the alkaloids. Repeat this procedure on the seeds until no more residue is obtained. This residue contains the natural alkaloids which are similar to LSD, as well as other plant products and impurities.

Add 100ml petroleum ether to 100g finely ground seeds and let soak about two days. Filter, discard and let seeds dry. Add 100 ml methanol to the seeds let soak about two days. Filter, repeat extraction with another 100ml methanol and evaporate the methanol extracts in vacuum. This yellow residual oil contains the alkaloids.

ERGOT ALKALOID HYDROLYSIS

NOTE about ergot. In order to make LSD, lysergic acid is needed. This can sometimes be obtained, but generally one of the lysergic acid containing ergot alkaloids such as ergotamine is more readily available. Ergot is the dried sclerotium of various s pecies of fungi which infect rye (and other grasses) leading to the formation of large purple growths in place of the rye grains. These growths are collected,dried,powdered and the alkaloids extracted. Ergot is mainly produced in Europe (especially Switze rland). Some is grown in the

USA, mainly for the use of ergotamine and related compounds in medicine (terminating migrane headaches etc) Many of the ergot alkaloids are deriatives (amides) of lysergic acid. Unfortunatly these compounds have little halluci nogenic activity and it is necessary to hydrolyze (split with water) off the amide, producing lysergic acid and to synthesise a different amide with greater psychadelic activity. This hydrolysis can be done with any of the following co

mpounds or a mixture of them : ergometrine , ergine , ergotamine , ergosine , ergocristine , ergokrytine , ergonovine (ergometrine) , and methysergide (Sansert). When -ine is added to the name (eg- ergotaminine) this indicated the isomers which will produ ce inactive iso-LSD. A conversion process is detailed below.

Dissolve 20mg of the alkaloid (previous extraction) in 200ml 1M KOH in methanol (ie. dissolve 56g KOH pellets in 1L 100% methanol) in a 1L heavy walled vacuum flask and evaporate the methanol in vacuum at room temperature. To prevent flask from cooling, thus prolonging the evaporation time place flask in a pan of water maintained at room temperature by gently heating or warm water running through. Add 400 ml 8% KOH in water to the residue and boil for one hour (under N2 if possible-this can be done by filling the flask with an N2 stream and loosely stoppering or by allowing a gently stream of N2 to flow through during heating. Cool, acidify with dilute sulfuric acid and shake in separatory funnel with 1L ether. Discard the upper ether layer and filter the aqueous suspension of lysergic acid(I) in vacuum. Wash precipitate with 20ml dilute sulfuric acid. To recover the small amount of (I) in solution remaining in solution, basify the Na carbonate and bubble CO2 through it. Filter and add precipitate to first batch. Some isolysergic acid will remain in solution and can be precipitated by adding 10% HNO3. It can be converted to (I) by adding 3ml 10% KOH for each 0.1g acid, boiling on steam bath for one hour under N2(if possible) and precipitating by acidifying with glacial acetic acid. Maximum yield is about 9g (I) for 20g ergotamine(alkaloid).

PURIFYING

About 20% of (I) will be isolysergic acid(non psychoactive) and it can be isomerized to (I) by the above procedure.
Purifying is not nessecary but can be done to improve quality as follows..

Dissolve 9g (I) in 20ml NH4 OH, filter and concentrate in vacuum at room temperature to precipitate (I). After filtering, the grey crystals can be further purified by dissolving in boiling water and cooling in ice bath to precipitate (I). Melting point (point it decomposes at) is about 240¡C

Alternatively the dark coloured (I) resulting from hydrolysis can be shaken with 2 X 400ml 2 M NH4 OH in ethanol and the combined extracts evaporated in vacuum to give (I). Dissolve the remaining residue in 500 ml hot methanol, cool to 0¡C and filter out the (I).

Coloured impurities can be removed by shaking solution with decolourizing carbon and filtering.

MAKING LSD

LSD FROM LYSERGIC ASID

Dissolve 13.4g dry (I) in 250 ml dry dimethformamide and cool to 0¡C. Add cooled solution of 3.4 ml 0.35 M methanesulfonic acid anhydride in dry dimethylformamide. After thirty minutes at 0¡C add 14.6g (20.4 ml) diethylamine (DEA) and keep at 0¡C for o ne hour. Evaporate in vacuum to get LSD.

Dissolve 5.3g dry (I) in 125 ml acentonitrile (or dimethylformamide or proprionitrile) and cool to -20¡C (freezer or dry ice-acetone or ethanol mixture). Add 8.82g trifluroacetic anhydride in 75 ml acetonitrile cooled to -20¡C carefully. Let stand at - 20 for 1* hours or until all (I) dissolves. Then add 7.6g DEA in 150ml acetonitrile and let stand at room temperature in dark two hours. Evaporate in vacuum to get LSD.

Dissolve 0.536g (I) in 10 ml freshly distilled POCL3; stir and add 416mg powdered, freshly sublimed PCl5. Hold two minutes at room temperature, two minutes at 90¡C, and evaporate in vacuum. Extract the residue with hexane to give the lysergic acid chlo ride-HCl (can also extract the reaction mixture with hexane instead of evaporating in vacuum). Alternatively use 6ml POCl3 and 240 mg SOCl2 and heat three minutes at 90¡C to get the acid chloride. To 5g of the acid chloride add 1.4ml DEA in 50ml methane c hloride and cool to 0¡C. Stir and add 27.5 ml pyridine and stir * hour at 0¡ . Warm to room temperature and stir 1* hours. Evaporate in vacuum to get LSD.

To a suspension of 13.4g dry (I) in 800ml dry dimethylformamide (DMF) in a 2L vacuum flask at 20¡, add a solution of 8.9g N,N'-carbonyldiimidazole in 250ml DMF and stir at 20¡ in dark for * hour. Add a solution of 4g DEA in 50ml DMF and let stand 2 hou rs at 20¡;then purify or dissolve residue in 2*L 2% tartaric acid; NH4OH and extract with a 9:1 solution of ether:ethanol. Dry and evaporate in vacuum to get LSD.

Add 1L dimethylformamide (freshly distilled if possible) to dry flask fitted with stirrer, ice bath, dropping funnel and condenser, both protected from water by Ca chloride drying tubes. Add dropwise with stirring over 4-5 hours at 0¡ 0.21bs (90.7g) So 3 (sulfuric anhydride, available as Sulfan from Allied Chem Co.) if precipitate forms, stir until it dissolves. Sulfan may be made in larger amounts and is good for several months if kept dry and cool. Molarity of fresh SO3-DMF reagant should be about 1M, but for precise determination add a little water to aliquot and titrate with standard NaOH to phenolphthalein end point. Add 6.45g dry (I) (or 7.15g (I) monohydrate) and 1.06 LiOH hydrate to 200ml methanolin a 1L vacuum flask and evaporate in vacuum. Dis olve residue in 400ml DMF at about 15mm Hg through a twelve inch column packed with glass helices or other material. Cool to 0¡ and rapidly add 50ml SO3-DMF solution (1 M). Stir at 0¡ for ten minutes and add 91.5g (12.9ml) DEA and stir ten minutes. Add 400ml water, stir and add 200ml saturated NaCl. Extract the LSD by shaking with several 500ml portions ethylene dichloride (can use indole test to show completeness of extraction). Combine

extracts (lower layor in seporatory funnel) and dry, evaporate in vacuum to get LSD.

To a reflexing slurry of 3.15g dry (I)(or monohydrate) in 150ml CHCl3 add 0.1 mole of the amine in 25ml CHCl3 and 2ml POCl3 simultaneously from seperate dropping funnels over 2 to 3 minutes. Reflux 3 to 5 minutes more till a clear amber solution result s. Cool to room temperature and wash with 200ml 1m NH4OH. Dry and evaporate in vacuum(below 40¡C). Can dissolve in the minumum amount of methanol. Filter and wash crystals with cold methanol and acidify with a fresh solution of 20% maleic acid in methanol . Filter and wash crystals with cold methanol to get the LSD or other amide. This method works with a wide variety of amines. For LSD itself, the POCl3 can be added first. The yield is about 50%.

PURIFYING

To purify your extracted LSD dissolve the residue in 150ml CHCl3 and add 20ml ice water. Pour into *L seperatory funnel and drain out the lower CHCl3 layer into a beaker (after shaking). Add 50ml CHCl3 to funnel, shake and drain bottom layer into same beaker. Repeat with 3 X 50ml CHCl3 and discard the water. Extract the combined CHCl3 extracts with 4 X 50ml ice cold water and dry, evaporate in vacuum the CHCl3 to get 3.5g d-LSD. This is composed partly of inactive d-iso-LSD which can be recovered and c onverted to d-LSD as follows: dissolve the residue in 120ml benzene and 40ml CHCl3 (or 200ml methanol), add tartaric or maleic acid and shake to precipitate mainly d-LSD (add a little ether and cool in refrigerator several days if nessecary to ensure comp lete precipitation)) Evaporate in vacuum the solvent to get d-iso-LSD. Ad 50ml ethanol and 5ml 4N KOH per g iso-LSD and let stand at room temperature for two hours, evaporate in vacuum (or extract with CHCl3 as above) to get LSD.

SYNTHESING LSD

Another way to go about it is to synthesise LSD entirely as I will now detail.

SYNTHESIS OF 2,3-Dihydrolysergic acid

Condense methyl-6-methyl-nicotinate and 5-Br-isatin by fusion at 180¡ to get 57% yield of (I) (I) in boiling glacial acetic acid is treated portionwise with powdered zinc and refluxed one hour to get (II). Treat (II) with NaBH4-BF3 (in ether) in tetrah hydrofuran as above to give (III) which when treated 24 hours with acetic anhydride gives (IV). Treat (IV) with methyl iodide in methanol-acetone in a Carius tube to get (V) which is reduced with KGH4 in aqueous methanol to get (VI). Treat (VI) with NH3 co ntaining NaNH2 for one hour to get 2,3-dihydrolysergic acid (VII) which can be converted to 2,3-dihydro-LSD which is about ten times less active than LSD. (VII) can be converted to lysergic acid prior to conversion to LSD, which will triple the yield in t erms of LSD activity.

Dehydrogenation may work for the next process and also may work for converting 2,3-dihydro-LSD into LSD.

LYSERGIC ACID FROM 2,3-Dihydrolysergic acid

To synthesise Lysergic acid from 2,3-dihydrolysergic acid dissolve 4g (VII) in 78ml 1.5% KOH and reflux five minutes (under N2 if possible). Add 8.5g Na arsenate hydrate and 16g Raney-Ni (wet [deactivated by boiling in xylene suspension]) and reflux tw enty hours (N2 if poss). Filter, precipitate lysergic acid by taking pH to 5.6 with HCl; filter and wash precipitate with water to get 1g lysergic acid. Evaporate in vacuum the filtrate to get more product.

NB: also see COMPLETE SYNTHESiS OF LSD for another method..

LSD VIA THE Hydrazide

Add 1.16g ergotamine.HCl to 4ml anhydrous hydrazine and heat one hour at 90¡. Add 20ml water and evaporate in vacuum. (Purify by adding ether and aqueous tartaric acid, basify the aqueous phase and extract aqueous phase with CHCl3 to get mainly d-iso- lysergic hydrazide (I) (Purify; chromatograph on alumina and elute with 0.5% ethanol in CHCl3)). To 1g (I),finely ground, in 40ml 0.1N ice cold HCl add with good stirring at 0W 4ml 1N Na nitrite. Quickly over 2-3 minutes add 40ml 0.1 N NaHCO3 and extract with 100ml ether, then 50ml ether. Wash ether with water and dry and evaporate in vacuum at 10¡.Dissolve the resulting yellow azide in about 5ml diethylamine(DEA) at 0¡ and heat one hour at 60¡ in a bomb(sealed metal pipe). Let stand several hours and ev aporate in vacuum to get about 0.7g d-LSD and 0.15g d-iso-LSD (convert as above). Alternativly the DEA can be added to the cooled ether solution of the azide and let stand several hours or overnight at room temperature in the dark in a vented flask.

LSD IDENTIFICATION

There are a few ways to test for LSD presence and strength..

LSD fluoresces under an ultraviolet light (black light), but so do many other compounds.

As LSD is an indole deriative it shows positive to these indole tests (which will also show DMT, psilocybe etc.)

KELLER TEST

Add a little of the powdered substance (0.2mg) to 1ml glacial acetic acid containing 0.5% FeCl3; layer underneath with 1ml concentrated sulfuric acid and shake. The colour varies with the indole. (Olive green - psilocin ; Red-Violet - psilocybin)

VAN URK TEST

Prepare Van Urk reagent by adding 0.5 g p-dimethylaminobenzaldehyde, 100ml water, 100ml concentrated sulfuric acid. Dissolve 1mg substanec in 1ml ethanol and mix with 2ml Van Urk reagent and illuminate for 10 minutes with an UV lamp (black light). (Psil ocin - blue-grey ; Psilocybin - red-brown)

QUICK TEST

Saturate strips of filter paper with a 2% p-dimethylaminobenzaldehyde in 45% ethanol; air dry and store in tightly stoppered amber bottles (or in dark), they will last several months. Put a little of the suspect substance in a few drops of

ethanol (gin may do as a control), wet a filter paper strip in this and allow to dry. Put one drop concentrated HCl on the dried paper (dont let it touch anything). Alternativly, the powder can be placed directly on the strip and the HCl dropped on it. A violet red or violet blue indicates indole deriatives such as LSD. With DMT or psilocybin the colout is redder. The colour must be observed soon after adding the HCl as it rapidly changes.

COMPLETE SYNTHESIS OF LSD

Mix 32.8g (0.217M) methyl-6-methylnicotinate (other alkyl groups can replace either methyl group) with 45.2g(0.2M) 5-bromoisatin (apparently 4-Br or 4 or 5 Cl isatin will also work) in a 250ml flask at 100¡ in an oil bath and raise the temperature to 1 70¡ and let react for 70 mins. Cool and then grind the solid as fine as possible in a mortar. Recrystallize from 150ml dimethylformamide and wash with ether to get 40g (57%)
methyl-(5-bromo-3-isatylidene)-6-methyl-nicotinate (I). Suspend 10g(I) in 250ml g lacial acetic acid and heat to boiling. Add in small portions over 30 mins excess powdered zinc. Reflux one hour, filter and evaporate in vacuum and recrystallize the residue from dioxane to get 9.7g(95%) methyl(5-bromo-2-oxindol-3-yl)-6-methylnicotinate (II) to get a suspension of 18g dry NaBH4 in 300ml dry tetrahydrofuran add with stirring at 0¡ over 30 mins about 75g BF3 etherate. Stir 3 hours at 0¡, add 18g (II) and heat exactly 20 mins at precisely 22-24¡. Add carefully 150ml concentrated HCl while cooling. Add 200ml water and stir 12 hours. Basify, extract the product with ethyl acetate and dry,evaporate in vacuum to get 11g of residue which recrystallizes from methanol to give methyl(2,3-dihydro-5-bromo-3-indolyl)
-6-methylnicotinate (III)

The following step may be unnessecary but it gives stability to (III). The acetyl group can be split off at the end of the synthesis, but is unnessecary since the 1-acetyl-LSD is as active as LSD.

Treat 12g (III) at room temperature for 24 hours with acetic anhydride then hydrolyze and extract to get 11.5g residue which is ground in petroleum ether and recrystallized from cyclohexane (can chromatograph on alumina and elute with petroleum ether t o was out an oil, then with benzene containing 5% ethyl acetate to elute the product) to give
methyl-(1-acetyl-2,3-dihydro-3-indolyl)-6-methylnicotinate (IV). Heat 5g (IV), 12.5ml acetone, 12.5ml methanol and 1.8ml methyl iodide for 18 hours in a Carius t ube at 70¡-80¡ C. Cool, filter, wash with acetone and recrystallize from methanol to get
methyl-(1-acetyl-2,3-dihydro-5-bromo-3-indolyl)-1,6-dimethylnicotinate iodide (V). To 9.4g (V) in 250ml water and 250ml methanol at 35¡ add over 5 mins 2.9g KBH4 and stir 10 mins. Add 2.9g more KBH4 and stir 30 mins. Evaporate in vacuum and extract the residue with methylene chloride to get about 6.2g oily mixture containing about 2g of the d isomer (can seperate by chromatography if desired) of
methyl-(1-acetyl-2,3-dihydro-5-bromo-3-indolyl)-6-methyl-1,2,5,6-tetrahydronicot-inate
(VI)

To a suspension of finely powdered NaNH2 (6.1g) in 2 litres dry ammonia, add with stirring 8g (VI) in 50ml dry tetrahydrofuran. Stir one hour, add NH4Cl and evaporate the ammonia as fast as possible in a nitrogen stream. Extract at pH 8 with methylene chloride to get 6g (can chromatograph on 300g silica gel and 250g celite and elute with

98% benzene-2% absolute ethanol and evaporate in vacuum) or methyl-1-acetyl-2,3-dihydro-lysergate (VII)

One method of dehydrating (VII) is above under LYSERGiC ACiD FROM 2,3-dihydrolysergic acid and another follows.

Warm to dissolve 1.5g 2,3-dihydro-LSD in 5ml acetone, 40ml water and 40ml saturated NaHCO3. Cool to 20¡ and add all at once with vigourous stirring 2.46g potassium nitrosodisulfonate dissolved in 90ml water and 10ml saturates NaHCO3. After 1 min, extra ct 7 times with ethylacetate, wash the combined extracts with water, dry and carefully remove solvent to get a mixture of 12-OH-LSD, LSD and starting material which can be chromatographed to give about 0.2g 12-OH-LSD.

The following method of converting (IV) to the diethylamide (which can probably be used in place of (IV) to give the diethylamide of (V), (VI) and (VII)) will probably also work admirably for (VII) or lysergic acid.

Reflux 0.5g (IV) with 0.5 KOH in 30 ml methanol for 4 hours. Evaporate in vacuum and add water to the residue. Adjust the pH to between 5 and 6 and filter or centrifuge to get 0.3g of the free acid. Suspend 1.8g of the acid in 125ml chloroform, cool to minus 5¡ and add 0.5g triethylamine, then 0.6g ethylchloroformate and stir 45 mins. Add 2 ml diethylamine and stir 3 hours at room temperature to get, after the usual workup 1g of the diethylamide (recrystallize from benzene)

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